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MODIFICATIONS IN THE VENETIAN TURPENTINE METHOD.

By C. M. Hopfinger.

Heretofore in working with the Venetian Turpentine method, fused calcium chloride only has been used as the drying agent in the concentration of the turpentine. There are however, other substances that have greater affinity for alcohol and alcohol vapor than calcium chloride. Definite compounds of alcohol and calcium chloride, as also of alcohol and zinc chloride are formed in which the organic substance plays the role of alcohol of crystallization, the double compounds being analogous to those formed with water when the latter takes the part of water of crystallization. Moreover, just as these later substances liquify in their own water of crystallization and absorb even more than corresponds to the definite compounds, so alcohol behaves towards the haloid salts of zinc and calcium. Zinc nitrate would serve perhaps as well as the chloride unless oxidation products were formed. Another compound absorbing alcohol in the same manner as these mentioned is barium hydroxide.*

Sulphuric acid behaves in a different way, absorbing alcohol

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* Some of these alcoholates, or in some cases ethylates, are the following, $LiCl\ 4C_2H_5(OH)$ with wood alcohol $LiCl\ 3C_2H_5(OH)$, $Mg\ Cl_2\ 6C_2H_5(OH)$, $Mg\ (NO_3)_2\ 6C_2H_5(OH)$, $Na_2S\ C_2H_5(OH)$, $CaCl_2\ 4C_2H_5(OH)$, also $CaCl_2\ 3C_2H_5(OH)$ when dried over H_2SO_4. $SnCl_4\ 2C_2H_5(OH)$ when heated gives off ether $(C_2H_5)_2O$, ethyl chloride, C_2H_5Cl; a compound $C_2H_5O\ SnCl_3 + C_2H_5(OH)$ is formed also. $TiCl_4$, $PtCl_4$, SeO, $AsCl_3$, $SbCl_5$ $CuSO_4$, take up one molecule of $C_2H_5(OH)$ to form a compound. KOH forms the compound $KOH\ 2C_2H_5(OH)$.

BaO and CaO form compounds which change to the ethylates, $(C_2H_5O)_2Ba$, etc. Wood alcohol acts somewhat similarly. I have not been able to discover whether a definite crystallizable compound of zinc chloride and alcohol has been isolated.

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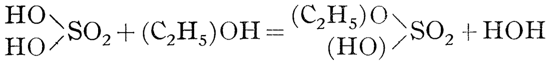
† July 15, 1912.—Pages 249 to 264.

with the formation of ethyl sulphuric acid, according to the equation,



$$\begin{matrix}HO\\HO\end{matrix}\!>SO_2 + (C_2H_5)OH = \begin{matrix}(C_2H_5)O\\(HO)\end{matrix}\!>SO_2 + HOH$$

Barium hydroxide seems to have but slight attraction for large quantities of alcohol and may be omitted from consideration.

Wood alcohol is also thus absorbed by all the above mentioned substances and hence "Synthol" substituted for absolute alcohol of late, because cheaper, can be as readily used in the Venetian Turpentine method with any of the above-mentioned chemicals. One of the best drying agents is zinc chloride. Although at first this does not seem likely, because the calcium chloride is far more porous, practical experiments show that this drawback is quite overcome by the greater attraction of the zinc chloride for the alcohol. When comparing the value of the zinc chloride with calcium chloride in this respect the absorption is more rapid in the case of the former. Other experiments show too, that a mixture made by fusing the zinc chloride and the calcium chloride together, worked better than the calcium chloride; but evidently not because of the increase of porousness, as this fused mixture is anything but porous.

Again, it was found that a mechanical mixture of the separately fused zinc chloride and calcium chloride also exceeds the calcium chloride as a drying agent. Sulphuric acid because of its affinity for alcohol appeared to have drying powers equal to the above-mentioned substances, but proved impracticable, because of its destructive effect upon the stain.

Results show that zinc chloride alone, or in combination (with calcium chloride), and sulphuric acid, increase the rapidity of absorption by one-half the time required when using calcium chloride. The greater the free surface of the drying agent, the more closely the vessel is stoppered, and the higher the temperature, the greater will be the available vapor tension of the alcohol, and therefore the more rapid the absorption.

The following table shows the time, as exact as could be determined, for the concentration of the turpentine, under the conditions which follow. In table No. 1, Hempel's vacuum dessicator was used, without applying diminished pressure, of course, and in table No. II, large covered crystallizing dishes. Through the whole series of experiments the remainder of the conditions were the same. Volume of Hempel's dessicator, 4200 cc; volume of crystallizing dishes, 2450 cc; amount of 10 per cent. Venetian Turpentine for each experiment, 25 cc; average free surface of the turpentine, 16sq. cm; and average temperature, 25°C. The amount of drying agent in each case was determined by the molecular weight of substance, 880 gm. calcium chloride being taken, and in case of the other agents an amount relatively proportionate.

These drying agents were not changed after each concentration, but kept for the whole series to ascertain the relative falling off in value after concentrating a number of the solutions. In the tables, the Roman numerals signify the successive turpentine preparations placed in the drying vessel, all of the same volume at start and finish, and following each other immediately in point of time. The figures in each column mark the time required for each concentration.

TABLE NO. I. (Dessicator, air-tight).

Order of Concentration	$ZnCl_2$ and $CaCl_2$ fused together	Machanical mixture of $ZnCl_2$ and $CaCl_2$ fused separately	Fused $ZnCl_2$	Fused $CaCl_2$	H_2SO_4
I.	24 hours	32 hours	25 hours	41 hours	32 hours
II.	35 "	41 "	39 "	73 "	40 "
III.	44 "	46 "	45 "		44 "
IV.	47 "	49 "	49 "		
V.	51 "	52 "			

TABLE NO. II. (Dessicator not air-tight).

Order of Concentration	Fused $ZnCl_2$	Fused $CaCl_2$	H_2SO_4
I.	40 hours	58 hours	37 hours
II.	44 "	75 "	41 "
III.	47 "	90 "	45 "
IV.	50 "		

From the above data we can satisfactorily conclude that for all practical work in the Venetian Turpentine method, either zinc chloride, or a mixture of zinc chloride and calcium chloride fused together, or a mechanical mixture of these two separately fused is better by far than calcium chloride. These three reagents have about the same drying powers, nearly equaling each other after sixth or seventh time used, although differing a little more at the beginning. Ten concentrations, according to conditions already given, seem to be the limit of practicability, since at this stage the reagents have liquified to such an extent, that the time for concentration exceeds the time required to fuse the drying substances again and begin a new series of concentrations.

In the case of sulphuric acid the mere concentration is satisfactory, but at the end of the process, the stain has been almost wholly destroyed. Perhaps for the acid stains this difficulty would be removed, yet on the whole it would hardly be desirable since other agents of equal drying power can be substituted.

Upon examination of the material when the experiment is completed, no evidences of plasmolysis can be detected, and this action might be suspected because of the short time required in the process. Throughout this experiment, Cladophora and Oedogonium were used with equal success. In estimating the time required by this shortened process, it was found that a specimen can be brought up ready for use in practically two days.

In preparing material by the Venetian Turpentine method, much time and trouble can be saved, if after killing with chromo-acetic acid and washing well, the specimen is brought up in the alcohols, in 10, 20, 35, 50, 70, 85 and 95 per cent. successively, instead of using the glycerine process, incurring the risk of traces of glycerine in the material, preventing proper staining. The time is hereby also lessened, as a few minutes is sufficiently long for the specimen to remain in each of the graded alcohols, when filamentary algae and the like are used.

If at this stage the specimen is placed in the 10 per cent. Venetian Turpentine, without previously being stained, as has been heretofore considered necessary, and then a given amount of the concentrated alcoholic solution of the Magdala Red added to the turpentine, and the whole placed in the drying vessel, the material will in the end be perfectly stained, the process of staining going on while the turpentine is concentrating. In performing these experiments Magdala Red alone was the stain used, and ten drops was found to be the minimum amount required in each experiment.

In trying to double stain, using Magdala Red and Aniline Blue, by pouring the concentrated alcoholic solutions of the stains together, then introducing the specimen, I succeeded a few times out of many trials, showing that generally the successful double staining by the above method is accidental, yet anticipating practical success, should we find the exact proportion according to which the stains should be mixed, even going so far as to insure success by the shortened process, recommended by the above experiments, in the case of the Magdala Red.

As to the reagents, an apparent objection to the use of fused zinc chloride instead of calcium chloride may be that the former on being fused again after absorption of alcohol seems to allow this to come off with great difficulty, so that the mass chars to a blackish substance, because of separation of carbon. After second fusion the zinc chloride does not seem to reabsorb alcohol as well as before, possibly because basic zinc chlorides are formed and some of the chlorine goes off as ethyl chloride (C_2H_5Cl). Addition of hydrochloric acid to decompose these basic compounds supposed to be found does not improve matters much.

The fused mixture of zinc chloride and calcium chloride in equal parts does not behave in this way; no charring whatever is noticed no matter how high the temperature is raised in fusion. Since moreover, the mixture is also more active we may conclude that it is the best dealcoholizer of them all. Whichever of these salts or mixtures of them is used, the fusion after the first absorption of alcohol may be made in either an iron or a porcelain vessel. Zinc chloride and mixtures of it show a tendency to absorb iron from the vessel during fusion, this iron appearing to be held in the colloidal form. This however does not deteriorate the absorptive power, though the fused mass appears to be steel gray in color. Care should be exercised to prevent the escaping alcohol vapors from catching fire in fusion, as charcoal is deposited, giving the preparation an unsightly appearance.

ERIGERON IN NORTH DAKOTA.

By J. Lunell.

In the year 1818 Nuttall published in Vol. II. of "The Genera of North American Plants" on p. 147 and 148 two new *Erigeron* species, *E. asper* and *E. glabellus*. During the last 25 years there has been considerable confusion as to what plants